Volume 3, Issue 8, August - 2025
ISSN (E): 2938-3781

Web of Agriculture: Journal of Agriculture and Biological Sciences
webofjournals.com/index.php/8

DETERMINATION OF INSECTICIDE RESIDUE LEVELS IN PLUM FRUITS

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Abstract
This scientific study was conducted to detect insecticide residues in plums grown in orchards across Uzbekistan during the years 2020–2022. The persistence of insecticides such as organophosphates, pyrethroids, avermectins, emamectins, and neonicotinoids - applied to control pests - was evaluated in the fruit. The analyses were carried out using gas-liquid chromatography (GLC) and high-performance liquid chromatography (HPLC) techniques. According to the results, insecticide residues were generally not detected in the fruit samples, while chlorpyrifos was present at levels below the established maximum residue limits (MRLS). The detection and monitoring of such residues highlight the importance of the rational selection and use of insecticides in ensuring human health protection.

Keywords: insecticide, plum, pesticide residue, gas-liquid chromatography, HPLC, biosafety, food safety, chlorpyrifos, dimethoate, cypermethrin, emamectin benzoate, acetamiprid.

Introduction

According to FAO/WHO Codex Alimentarius, international standards have been established for pesticide residues in food products, particularly for products intended for export. Export-oriented producers are required to comply with these standards by disclosing which insecticides were applied against pests on their crops.

The persistence of insecticide residues in fruits depends on several factors, including the chemical nature of the compound, the structural composition of the fruit, the dosage applied, and the timing of pesticide application.

In orchards, insecticides belonging to various chemical classes such as organophosphates, pyrethroids, avermectins, emamectins, and neonicotinoids -specifically **chlorpyrifos, dimethoate, cypermethrin, lambda-cyhalothrin, emamectin benzoate, and acetamiprid**-were evaluated for their residue persistence in fruit matrices.

Analyses were conducted using **Gas Chromatography (GC)** and **High-Performance Liquid Chromatography (HPLC)** techniques. The detection and quantification of pesticide residues play a critical role in the rational selection of insecticides and in ensuring consumer health protection.

Related Works

The determination of pesticide residue levels in fruits and vegetables plays a vital role in ensuring food quality and safeguarding human health. According to international data, approximately 2.5

Volume 3, Issue 8, August - 2025
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million tons of insecticides are produced annually worldwide, with about 27% of them being applied specifically to fruits and vegetables [1, 2].

Data from the United Nations Children's Fund (UNICEF) and the World Health Organization (WHO) indicate that pesticide poisoning accounts for around 300 million cases globally each year, with approximately 220,000 of these resulting in fatalities [3]. Taking this into consideration, in 2019, the Food and Agriculture Organization (FAO) declared the year 2020 as the "International Year of Plant Health" [4, 5].

Among insecticides, the most widely used are those belonging to the neonicotinoid class [6].

The level of pesticide residues in fruits varies significantly across different countries and regions, which is closely related to local pesticide application practices, regulatory standards, and agricultural management strategies [8]. In China, pesticides such as chlorpyrifos, imidacloprid, acetamiprid, carbendazim, and prochloraz are extensively used for the control of apple pests. Due to their high stability and water solubility, these pesticides tend to persist in the fruit [9].

Materials and Methods

Within the scope of the study, samples were collected from plum orchards to evaluate the presence of pesticide residues resulting from the application of newly developed chemical insecticides against pests. These samples were taken from trees treated with insecticides based on the following classes of chemical compounds:

- **Organophosphates**: *chlorpyrifos, dimethoate*
- **Pyrethroids**: *cypermethrin, lambda-cyhalothrin*
- **Emamectins**: *emamectin benzoate*
- **Neonicotinoids**: *acetamiprid*

For analytical procedures, internationally recognized analytical standards from *Sigma Aldrich* were utilized [7]. Residue detection of pesticides was carried out using modern physicochemical analytical methods, specifically **Gas Chromatography (GC)** and **High-Performance Liquid Chromatography (HPLC)** [10-11].

To identify insecticide residues in plum fruits, the samples were extracted using hexane and 96% ethanol. Subsequent analyses were performed via GC and HPLC methods.

To carry out the pesticide residue analysis, samples were collected at the ripening stage from both treated and untreated (control) plots in the orchard. The collected fruits were dried at room temperature in the shade for 6–7 days, and then ground into particles of 2–3 mm size.

From the dried samples, 20 g were weighed and placed into a 100 ml flask, followed by the addition of 50 ml of either hexane or 96% ethanol. The mixture was subjected to ultrasonic extraction in a water bath for 20 minutes and repeated three times. The extracts were then allowed to settle and filtered through a 0.45 µm pore-size membrane filter to prepare them for GC or HPLC analysis.

Standard preparation:

- For GC analysis, insecticide standards were prepared at a concentration of **1 mg/ml.**
- For HPLC analysis, standards were prepared at **0.01 mg/ml** concentration.

3.1 Gas Chromatography (GC) Analysis Conditions:

- **Instrument**: Shimadzu GC-2010 Gas Chromatograph
- **Column**: Rtx capillary column (30 m × 0.25 mm × 0.25 µm)

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- **Mobile phase**: Nitrogen gas, flow rate – 1.0 ml/min
- **Injector temperature**: 250°C
- **Detector**: FID detector, temperature – 300°C
- **Temperature program**:
  - Initial: 50°C, held for 2 minutes
  - Ramp: Increase at 15°C/min to 280°C
  - Final: Hold at 280°C for 5 minutes
- **Injection volume**: 1 µl
- **Injection method**: Manual injection

3.2 High-Performance Liquid Chromatography (HPLC) Analysis Conditions:

- **Instrument**: Shimadzu LC-20AP HPLC system
- **Column**: C18, 150 × 4.6 mm, 5 µm
- **Mobile phase**:
  - Phase A: Water with 0.1% phosphoric acid
  - Phase B: Acetonitrile
- **Elution mode**: Isocratic (A:B – 40:60)
- **Flow rate**: 1.0 ml/min
- **Detector**: UV detector, wavelength – 220 nm
- **Injection volume**: 20 µl

Results and Discussion

For each insecticide, standard fruit test samples were prepared, and after undergoing a specific pre-treatment process, analytical detection procedures were carried out.

Dried plum fruits of the cultivars Vengerka, Serbia, and Fortuna, along with a control sample, were subjected to extraction using hexane and ethyl alcohol. The prepared extracts were analyzed using gas-liquid chromatography (GLC) and high-performance liquid chromatography (HPLC), depending on the chemical nature of the active substances present. To determine the residual levels of the insecticides cypermethrin, chlorpyrifos, and dimethoate in the dried plum fruits, samples were extracted in hexane and analyzed using the gas-liquid chromatography (GLC) method. This analysis aimed to identify the presence or absence of these active substances in the fruit matrix.

For this purpose, dried plum samples of the Vengerka, Serbia, and Fortuna cultivars-previously treated with the aforementioned insecticides-were prepared. These samples underwent extraction in an ultrasonic bath, followed by solvent evaporation to concentrate the extract, which was then filtered through a membrane filter. The final extracts were analyzed using a GLC instrument.

Table 1 Pesticide formulations used against pests commonly found in plum orchards (2020–2022)

Class of Pesticides	Name of the Pesticide Product	Active Ingredient
Organophosphates	Nurell extrim 67% EC	*chlorpyrifos,*
	Bi-58 40% EC	*dimethoate*
Pyrethroids	Nurell extrim 67% EC	*cypermethrin,*
	Espada 35% SC	*lambda-cyhalothrin*
Emamectins	Proclaim Fit 45% WG	*emamectin benzoate*
Neonicotinoids	Karache extrim 27% WP	*acetamiprid*

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According to the gas-liquid chromatographic analysis results (Figure 1) of the cypermethrin standard (black line) and the samples of Vengerka (brown), Serbia (blue), and Fortuna (red) cultivars, the chromatographic peak characteristic of cypermethrin (t_a = 7.7 minutes) was not observed in any of the samples. This indicates that cypermethrin was not detected in the fruit content.

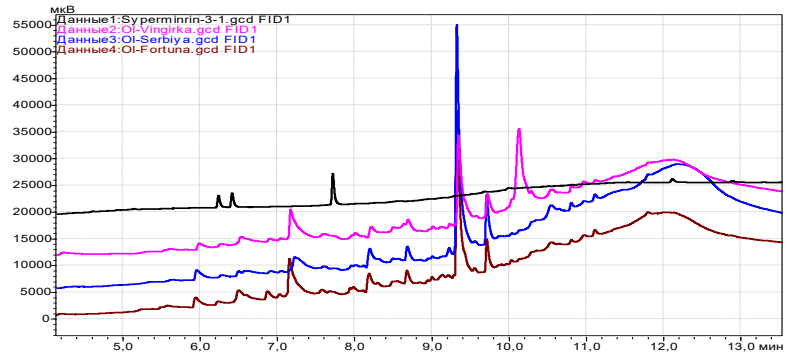

Figure 1. Chromatograms of the cypermethrin standard sample and plum extracts of the Vengerka, Serbian and Fortuna cultivars**.**

The standard sample of the pesticide chlorpyrifos and the extracts from dried plum fruits of the Vengerka (brown), Serbian (blue), and Fortuna (red) cultivars were comparatively analyzed using the gas-liquid chromatography (GLC) method.

According to the analysis results, a characteristic chromatographic peak of chlorpyrifos (t_a = 7.2 minutes) was detected in all three cultivars. This finding indicates the presence of chlorpyrifos residues in the analyzed plum samples (Figure 2).

The standard sample of dimethoate and the extracts of dried plum fruits - Vengerka (brown), Serbia (blue), and Fortuna (red) cultivars - were comparatively analyzed using gas chromatography (GC).

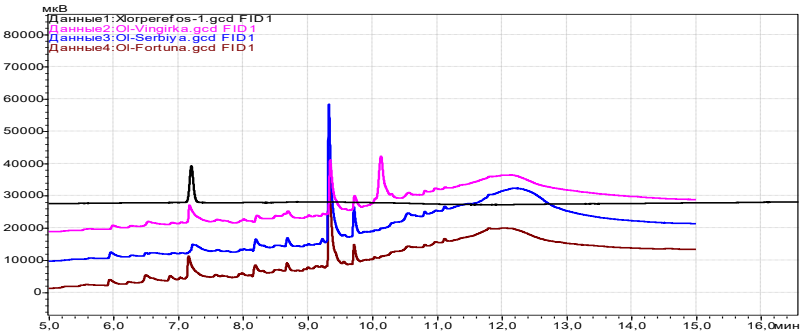

Figure 2. Chromatograms of the standard sample of chlorpyrifos and plum samples of the cultivars Vengerka, Serbia and Fortuna

According to the results, the chromatographic peak specific to dimethoate (t_a = 13.45 minutes) was not observed in the tested samples. This indicates the absence of dimethoate residues in the analyzed plum samples (Figure 3).

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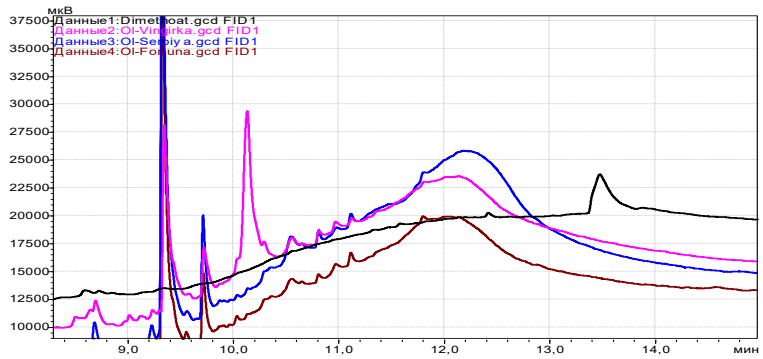

Figure 3: Chromatograms of Dimethoate Pesticide and Samples Belonging to the Vengerka, Serbian, and Fortuna Plum Cultivars

The analysis of insecticide residues in plum samples indicates that the degradation rate of chemical insecticides in the fruit varies depending on the class of compound. In the case of organophosphorus compounds, the pesticide residue was detected in amounts not exceeding the maximum residue limit (MRL) [12].

In contrast, pesticides based on neonicotinoids, emamectins, and other formulations degraded rapidly, with no detectable residue remaining in the fruit.

To detect the presence of emamectin benzoate, acetamiprid, and lambda-cyhalothrin pesticides in alcoholic extracts of dried plums, the high-performance liquid chromatography (HPLC) method was employed. This technique is known for its high accuracy and sensitivity, allowing the detection of even trace concentrations of pesticide residues.

Prepared samples were analyzed by comparing them with standard reference solutions of the aforementioned pesticides. According to the results, chromatographic peaks specific to emamectin benzoate, acetamiprid, and lambda-cyhalothrin were not observed in the chromatograms of the tested samples. This indicates the absence of these pesticide residues in the plum samples. The results confirm that the insecticides were applied in accordance with regulatory requirements and that their degradation or complete dissipation from the fruit had occurred by the time of analysis.

Conclusion

When insecticides used in plum cultivation are applied in accordance with regulatory standards, they either do not remain in the fruit or are present at residue levels below the established maximum residue limits (MRLS). Specifically, residue of the organophosphorus compound chlorpyrifos was detected in the fruit, indicating that its application requires careful and controlled use. Furthermore, insecticides based on neonicotinoids and emamectins were not detected in the fruit, as they degrade rapidly over a short period. This suggests that, when applied at recommended rates, these compounds can be considered safe for human health.

Importantly, the analyses conducted on samples from the Hungarian (Vengerka), Serbian, and Fortuna plum cultivars demonstrated that the amount of pesticide residues did not vary between cultivars. This indicates that the level of pesticide residues is primarily determined not by the plum variety, but by the type of insecticide used, the application technology, dosage, and timing.

Therefore, the rational selection of insecticides, along with their application in accordance with scientific recommendations and agro-technical standards, is a decisive factor in ensuring product

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safety. Such an approach not only safeguards human health and biological safety but also contributes to the production of export-oriented and competitive agricultural products.

Future Research Directions

Future studies should focus on:

It is essential to apply insecticides at lower application rates and to prioritize the Integrated Pest Management (IPM) approach when combating harmful insects that threaten yield quantity and quality in fruit orchards. Additionally, studying the persistence of insecticides in the soil and evaluating their impact on soil microflora is of significant importance.

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